

AD-A196 009

REPORT DOCUMENTATION PAGE

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1a. REPORT SECURITY CLASSIFICATION Unclassified		1b. RESTRICTIVE MARKINGS	
2a. SECURITY CLASSIFICATION AUTHORITY JUL 25 1988		3. DISTRIBUTION / AVAILABILITY OF REPORT Approved for public release and sale. Distribution unlimited.	
2b. DECLASSIFICATION / DOWNGRADING SCHEDULE		5. MONITORING ORGANIZATION REPORT NUMBER(S)	
4. PERFORMING ORGANIZATION REPORT NUMBER(S) ONR Technical Report No. 1		7a. NAME OF MONITORING ORGANIZATION Office of Naval Research Resident Representative	
6a. NAME OF PERFORMING ORGANIZATION UNIVERSITY OF WYOMING	6b. OFFICE SYMBOL (if applicable)	7b. ADDRESS (City, State, and ZIP Code) University of New Mexico Bandelier Hall West Albuquerque, NM 87131	
6c. ADDRESS (City, State, and ZIP Code) DEPARTMENT OF CHEMISTRY UNIVERSITY OF WYOMING LARAMIE WY 82071-3838	8a. NAME OF FUNDING / SPONSORING ORGANIZATION Office of Naval Research	8b. OFFICE SYMBOL (if applicable) ONR	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER N00014-87-K-0674
8c. ADDRESS (City, State, and ZIP Code) 800 N. Quincy Street Arlington, VA 2217	10. SOURCE OF FUNDING NUMBERS		
	PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO. R & T 41330190
11. TITLE (Include Security Classification) (u) Mass Measurements Using Isotopically Labeled Solvents Reveal the Extent of Solvent Transport During Redox in Thin Films on Electrodes			
12. PERSONAL AUTHOR(S) Steven J. Lasky and Daniel A. Buttry*			
13a. TYPE OF REPORT Technical	13b. TIME COVERED FROM 9/87 TO 6/88	14. DATE OF REPORT (Year, Month, Day) 1988 July 6	15. PAGE COUNT 9
16. SUPPLEMENTARY NOTATION Prepared for publication in the Journal of the American Chemical Society			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD	GROUP	SUB-GROUP	
		electrochemistry, quartz crystal microbalance, modified electrode, <i>signature</i>	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) Abstract - Thin films of nickel-ferrocyanide are grown at nickel electrode surfaces by electrochemical oxidative treatment. This report describes some new microgravimetric, quartz crystal microbalance (QCM) experiments using isotopically substituted solvents which detail the transport of solvent during the redox process of the Fe sites. The results show that solvent incorporation accompanies cesium ion expulsion during oxidation, and vice versa during reduction. The relationship between the numbers of moles of expelled cesium ions and incorporated water molecules indicates that considerable void space exists within the lattice of the reduced form of the metalocyanide film as a result of a 'discreteness of size' effect. To our knowledge, these experiments represent the first accurate, unambiguous measurements of solvent transport in thin films on electrodes. <i>key</i>			
20. DISTRIBUTION / AVAILABILITY OF ABSTRACT <input checked="" type="checkbox"/> UNCLASSIFIED/UNLIMITED <input type="checkbox"/> SAME AS RPT <input type="checkbox"/> DTIC USERS		21. ABSTRACT SECURITY CLASSIFICATION Unclassified	
22a. NAME OF RESPONSIBLE INDIVIDUAL Daniel A. Buttry		22b. TELEPHONE (Include Area Code) (307) 766-6677	22c. OFFICE SYMBOL

OFFICE OF NAVAL RESEARCH

CONTRACT NUMBER N00014-87-K-0674

R & T CODE 413301901

Technical Report # 1

Mass Measurements Using Isotopically Labeled Solvents Reveal the Extent of
Solvent Transport During Redox in Thin Films on Electrodes

Steven J. Lasky and Daniel A. Buttry*
Department of Chemistry
University of Wyoming
Laramie, WY 82071-3838

Prepared for publication in the Journal of the American Chemical Society

July 6, 1988

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Mass Measurements Using Isotopically Labeled Solvents Reveal the Extent of
Solvent Transport During Redox in Thin Films on Electrodes

Steven J. Lasky and Daniel A. Buttry^{*}

Department of Chemistry

University of Wyoming

Laramie, WY 82071

Abstract - Thin films of nickel ferrocyanide are grown at nickel electrode surfaces by electrochemical oxidative treatment. This report describes some new microgravimetric, quartz crystal microbalance (QCM) experiments using isotopically substituted solvents which detail the transport of solvent during the redox process of the Fe sites. The results show that solvent incorporation accompanies cesium ion expulsion during oxidation, and vice versa during reduction. The relationship between the numbers of moles of expelled cesium ions and incorporated water molecules indicates that considerable void space exists within the lattice of the reduced form of the metallocyanide film as a result of a 'discreteness of size' effect. To our knowledge, these experiments represent the first accurate, unambiguous measurements of solvent transport in thin films on electrodes.



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Transport of solvent during the redox reactions of thin films on electrodes has been identified as a possible influence on both the thermodynamic and kinetic aspects of their electrochemical responses (1). A variety of methods has been used in attempts to measure solvent content of these films, including ellipsometry (2,3) and profilimetry (4). However, those techniques which rely on measurement of thickness suffer from inability to deconvolute the contributions to swelling (or deswelling) from ion and solvent transport. Thus, the situation remains one in which speculation abounds, but accurate measurements are unavailable. In this Communication, we report on the application of the quartz crystal microbalance (QCM) technique to the determination of solvent transport during redox in thin films of nickel ferrocyanide (the nickel analog of Prussian Blue (5-12)) by comparing the difference in the total mass change (comprised of contributions from both ion and solvent transport) which results from use of isotopically substituted solvent. To our knowledge, these experiments represent the first accurate, unambiguous measurements of solvent transport in thin films on electrodes. It is especially significant that these measurements are made in the presence of simultaneous ion transport.

The QCM apparatus has been previously described (13-15). When used in conjunction with electrochemical measurements, it allows for the simultaneous determination of minute (multilayer to submonolayer) mass changes which accompany the electrochemical reaction with a mass sensitivity of 56.6 Hz/microgram/cm² in the present experimental configuration. Nickel films were deposited onto the QCM gold electrode either using a modified Watts bath (16) or by vapor deposition with identical results. The nickel ferrocyanide films were generated using the method of Bocarsly and coworkers (7), by maintaining the electrode potential at 1.2 V in a solution of 0.1 M KCl and 0.01 M K₄Fe(CN)₆ for the time required to obtain the desired film thickness. Conditions were precisely controlled to ensure uniformity of the deposited films, since this is crucial for the quantitative comparison of the QCM frequency change (which gives the mass change) to the electrochemical charge (17). FTIR microscopy (Mattson Cygnus 100 with Bach-Shearer microscope) was used to verify the film uniformity. Using a 10 micron spot size, the integrated intensity of the 2090 cm⁻¹ CN stretching band was measured at several points across the face of the QCM electrode, with special attention paid to the edge region. This procedure showed that the films were uniform to

within experimental error. Following film growth, the electrochemical and QCM responses of the nickel ferrocyanide film were examined in 0.1 M CsCl solutions prepared using either H_2O or D_2O . The films were electrochemically cycled for a sufficient period (usually ca. 10 minutes) to ensure that all of the K^+ ions from the growth solution had been exchanged for Cs^+ , as judged by the attainment of a constant QCM frequency for the reduced film. No interference from K^+ was observed after this procedure.

Figure 1 shows the results of such an experiment. Curves a and b in Figure 1A show that the cyclic voltammetric response is identical in these two solutions. The measured electrochemical charges are also identical, indicating that the same number of Fe redox sites are electroactive in the two solutions. The cyclic voltammetric response shown is typical of those reported for these films in Cs^+ containing electrolytes (6,7) with the exception of a somewhat larger peak to peak separation, a reminder of the sensitivity of these highly structured interfaces to the method of preparation(7). Curves a and b in Figure 1B show the QCM frequency response for these two solutions, collected simultaneously with the electrochemical data. The frequency increases during oxidation, and this increase is larger in the H_2O solution than in the D_2O solution. When the experiment was repeated in both D_2O and H_2O solutions of $CsNO_3$, identical results were obtained, providing unambiguous evidence that the observed frequency changes do not depend on the anion of the supporting electrolyte in any way.

That the frequency increases during oxidation is not unexpected, based on the earlier work of Bocarsly and coworkers (7,8). Their results indicated that the Fe(II) state of the films contained two cations per formula unit (i.e. $Cs_2Ni[Fe(CN)_6]$) while the Fe(III) state contained one (i.e. $CsNi[Fe(CN)_6]$), so that oxidation should cause the expulsion of one cation per formula unit to maintain electroneutrality. Thus, an increase in frequency resulting from the expulsion of the cations during oxidation occurs. In addition, the dependence of the frequency change on the mass of the solvent implied some degree of solvent transport during the redox event for these films. Quantitative comparison of the QCM frequency change with the electrochemical charge reveals the extent of each.

Exactly one cation should be expelled per electron removed from the film during oxidation. Direct comparison of the electrochemical charge and the QCM frequency change revealed that the mass loss (frequency increase) which

occurred during the oxidation was not sufficient to account for all of the expelled cations, i.e. a larger frequency change would have been observed if the only transport during the redox process were cation expulsion in the amount required to maintain electroneutrality. Thus, some mass must be regained by the film during or following cation expulsion to give the observed frequency change. If this mass were from solvent incorporation, then use of D_2O in place of H_2O would cause a detectable change in the observed frequency increase. Figure 2 shows a sample calculation for a typical film based on this hypothesis. Similar results were obtained for a variety of films with varying thicknesses. For this film, the calculation using the total anodic charge ($2.9 \times 10^{-3} \text{ C/cm}^2$) indicated that a frequency increase of 224 Hz should have been observed if the sole mass change were the expulsion of $3 \times 10^{-8} \text{ mole/cm}^2$ of Cs^+ . The observed frequency increases in D_2O and H_2O were 115 and 125 Hz, respectively. The discrepancies between the calculated and observed values in D_2O and H_2O were thus 109 and 99 Hz, respectively. (Given the signal to noise ratio of the measurement, the minimum detectable frequency difference is ca. 0.5 Hz.) If the entire discrepancy between the calculated frequency increase for Cs^+ and that observed experimentally were due to a net increase in the solvent content of the film, then the discrepancy for the D_2O case should have been 10% larger than that for the H_2O case because the molar mass of D_2O is larger than that of H_2O by 10%. This is exactly what was observed. The agreement is quantitative, and the requirement that the difference between the calculated mass gain due to D_2O and H_2O be 10% serves as an internal check of the veracity of the calculations and underlying assumptions. One such assumption is that D_2O and H_2O behave in chemically identical manners so that identical numbers of moles of each are transported during the two experiments shown in Figure 1. A most important assumption, crucial to the use of the frequency change in quantitative calculations (14,15), is that the film behave as a rigid layer so that the frequency change may be linearly related to the mass change. The excellent agreement presented above is unambiguous evidence of this. Feldman and Melroy (18) have previously reported rigid layer behavior for Prussian Blue films. In their study qualitative evidence for solvent transport during redox was observed.

The picture which emerges for the cesium case is that during oxidation some Cs^+ ions are expelled with a consequent influx of solvent to fill the void volume left within the lattice. During reduction, the species undergo

transport in the opposite direction, with complete reversibility of the mass changes. These data show that 3.2 moles of H_2O are incorporated when 1 mole of Cs^+ ions is expelled. Approximate molar volumes for the two species are $18 \text{ cm}^3/\text{mole}$ (from the bulk density of water) and $12 \text{ cm}^3/\text{mole}$ (from the ionic radius of Cs^+ of 0.169 nm given by Pauling (19)), so at first glance it seems that expulsion of one mole of Cs^+ should allow for accommodation of only 0.67 mole of H_2O . Thus, either the effective volume of Cs^+ within the lattice is considerably larger than its ionic radius would indicate, or the effective volume of a water molecule within the lattice is considerably smaller than it is in bulk water or some combination of the two. Since it is unreasonable to postulate a water density within the lattice which is enough larger (by a factor of nearly 5!) than the bulk value to account for the discrepancy, we attribute the majority of the effect to a much larger effective volume for Cs^+ . Geometric arguments based on the sizes of Cs^+ and H_2O in relation to the size of the octahedral cavity (10,20-22) can be made which are in agreement with this conclusion. Briefly, even though the Cs^+ cation does not completely fill the cubic cavity (which is 0.5 nm on a side (10,20-22)), it is just large enough to preclude the presence of water molecules. Thus, void space is left within the cavity which does not have the proper geometry to accommodate the incorporation of interstitial water. A more detailed description of this 'discreteness of size' effect will be reported in a future contribution, along with a discussion of the insight provided by such measurements of solvent transport into the remarkable sensitivity of the formal potential of these nickel ferrocyanide films to the identity of the presence of various alkali metal cations.

ACKNOWLEDGMENTS. We gratefully thank the Office of Naval Research for the support of this work.

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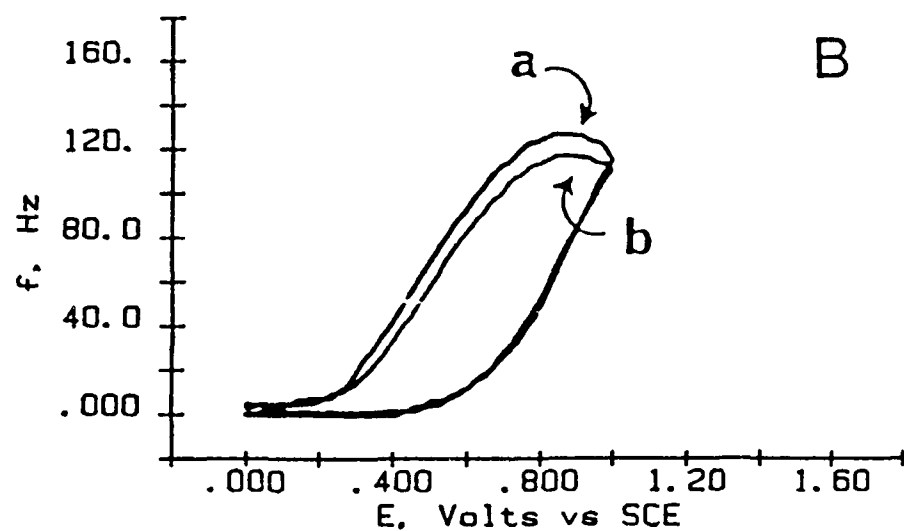
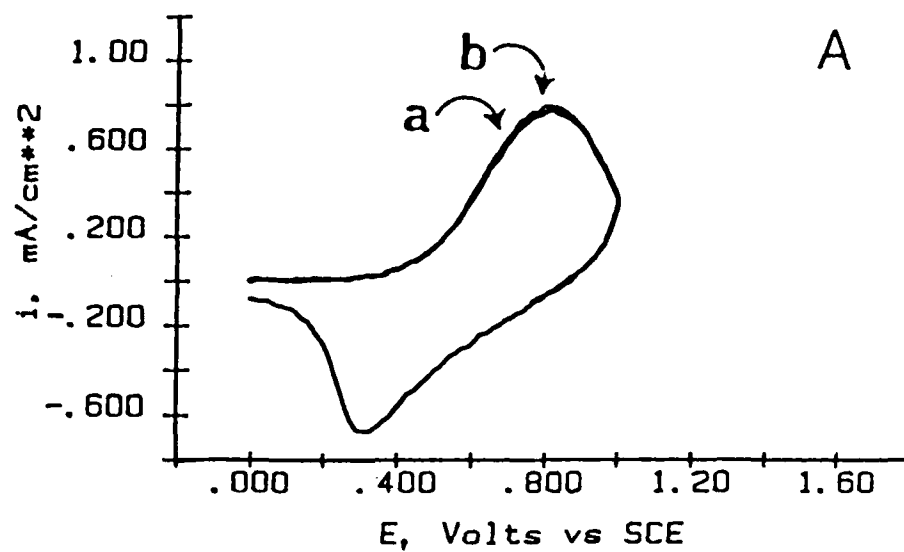
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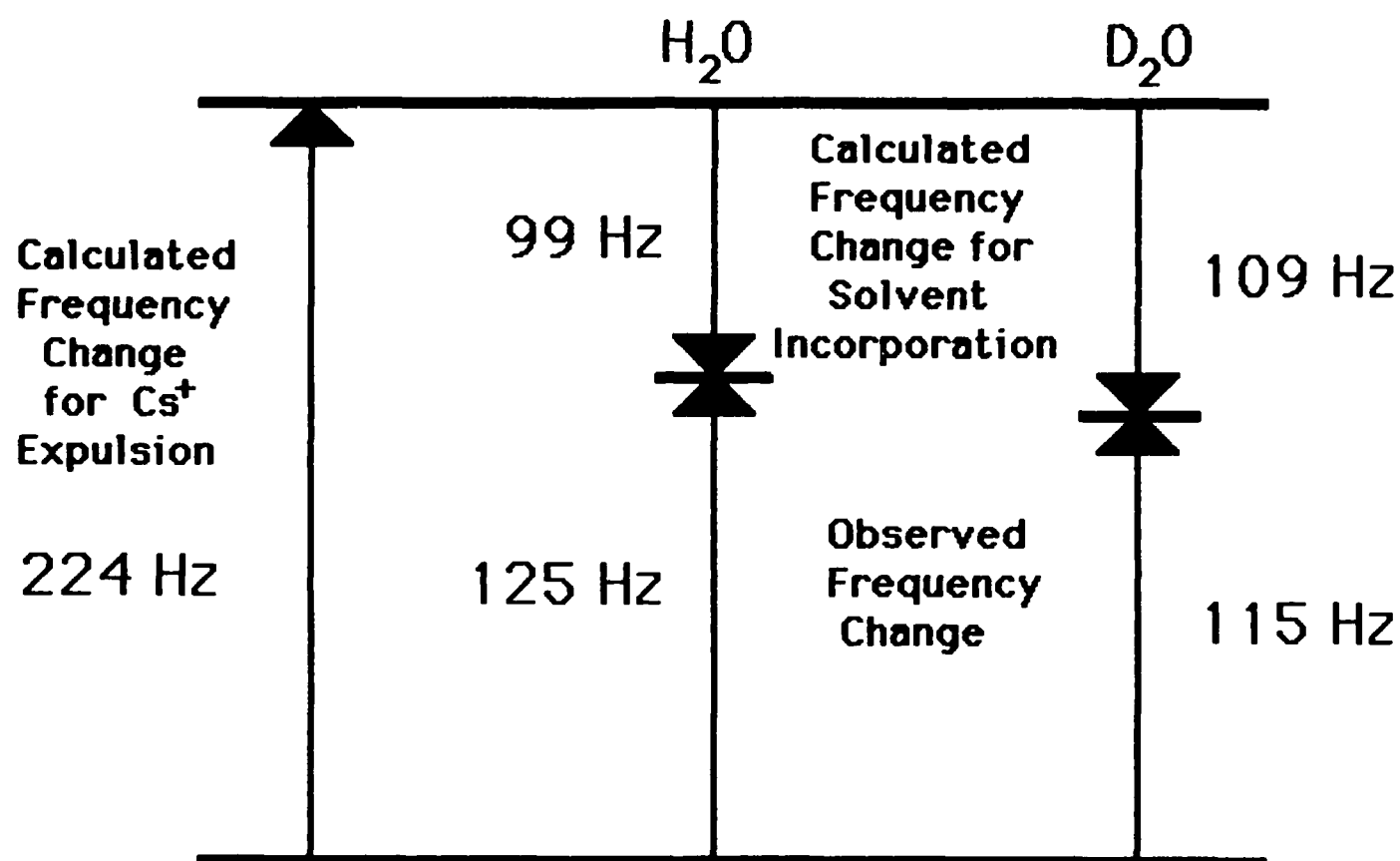
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FIGURE CAPTIONS

Figure 1 - A) a. Cyclic voltammogram of a nickel ferrocyanide film in 0.1 M CsCl/H₂O solution. Scan rate - 100 mV/s. b. Same as in (a) except that the solution contains D₂O instead of H₂O. B) a. QCM frequency response measured simultaneously with the cyclic voltammogram in (a). b. QCM frequency response measured with cyclic voltammogram in (b).

Figure 2 - Schematic representation of the frequency changes measured during the cyclic voltammogram in Figure 1. See text for details.





D/1113/87/2

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Institut für Technische Elektrochemie
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9 Getreidemarkt, 1160 Wien
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Dr. Henry S. White
Department of Chemical Engineering
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University of Wyoming
Laramie, Wyoming 82071

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